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*Indian Standard*

**METHODS FOR  
DETERMINATION OF  
STRENGTH OF BASIC DYES**

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**BUREAU OF INDIAN STANDARDS**  
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NEW DELHI 110002

# Indian Standard

## METHODS FOR DETERMINATION OF STRENGTH OF BASIC DYES

### 0. FOREWORD

**0.1** This Indian Standard was adopted by the Bureau of Indian Standards on 29 April 1988, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

**0.2** Basic dyes are generally marked by brilliance of shade and high tinctorial value. They can be applied on cellulosic fibres with the help of mordants. They are also used for dyeing and printing of protein fibres (wool, silk etc.) and polyacrylonitrile fibres as well as for topping of cellulosic materials dyed with direct dyes.

**0.3** Since the range of basic dyes for textile use is very wide and the quality of these dyes differ from manufacturer to manufacturer, it is very essential to know their strength so as to minimize cost-benefit ratio. This will also help to adjust recipes for dyeing and printing which minimize

variation in dyeing from one lot to the other and reduces chances of rejection of the dyed or printed material. Method 1 prescribed in this standard is based on the comparison of transmission of the solution of dyes under test with that of the standard dye whose strength is assumed to be 100 percent. It is suitable for dye solutions which do not scatter light and which obey the Bouguer-Lambert-Beer Law. Method 2 is based on the reduction of the dye with titanium trichloride and then calculating the strength of dye by the amount of titanium trichloride used up in the reduction.

**0.4** In reporting the results of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2 - 1960\*.

\*Rules for rounding off numerical values (revised).

### 1. SCOPE

**1.1** This Indian Standard prescribes two methods for the determination of strength of basic dyes.

### 2. TERMINOLOGY

**2.1 Depth of Shade** — The amount of dyestuff in g in the dyebath per 100 g of the fibre, expressed as a percentage.

**2.2 Strength of Dyestuff** — The percentage ratio of the strength of the dyestuff under test to that of the standard dyestuff, the strength of which is assumed to be 100 percent.

### 3. STANDARD DYESTUFF

**3.1** The standard sample of dyestuff against which the strength of dyestuff under test is evaluated, shall be as agreed to between the buyer and the seller.

### 4. QUALITY OF REAGENTS

**4.1** Unless otherwise specified, pure chemicals shall be employed in tests and distilled water (see IS : 1070 - 1977\*) shall be used where the use of water as a reagent is intended.

\*Specification for water for general laboratory use (second revision).

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

### 5. METHOD 1

#### 5.1 Apparatus

**5.1.1 Suitable Colorimeter** — capable of measuring transmission from 400 to 700 nm.

**5.1.2 Volumetric Flask** — 250 ml capacity.

**5.1.3 Weighing Balance** — with an accuracy of 1 mg.

**5.1.4 Watch Glass** — for weighing the dye.

**5.1.5 Glass Rod** — For preparing dye solution.

**5.1.6 Pipette** — With an accuracy of 0.05 ml.

#### 5.2 Reagent

**5.2.1 Methylated Spirit**

#### 5.3 Procedure

**5.3.1** Weigh accurately 250 mg of the standard dye (see 3.1). Paste the weighed amount of dye with 5 - 10 ml of methylated spirit in a volumetric flask. Pour 50 ml of boiling water with constant stirring. Cool the contents and make the solution up to 250 ml. Shake the flask thoroughly so as to homogenize the contents.

**5.3.2** Pipette out 0.1, 0.2, 0.3, 0.4 and 0.5 ml of solution prepared in 5.3.1 in separate test tubes, and make the total volume in each test tube to 100 ml before measurement.

**5.3.3** Calculate the concentration  $C_s$  in g/litre of each dye solution by the formula:

$$C_s = \frac{X}{100}$$

where  $X$  is the volume of dye solution taken ( see 5.3.2 ).

**5.3.4** Measure the transmission of the dye solutions prepared in 5.3.2 on the colorimeter at wavelengths of minimum transmission (maximum absorption) and then record the transmission of all the dye solutions prepared in 5.3.2 at this selected wavelength.

**5.3.5** From values of transmission of dye solutions obtained in 5.3.4, calculate the optical density of each solution separately by the formula:

$$\text{Optical density} = \log \left[ \frac{1}{T} \right]$$

where  $T$  is the percent transmission.

**5.3.6** Plot a graph between the concentration of dye solution ( $C_s$ ) along  $X$ -axis against the optical density along  $Y$ -axis, which shall be a straight line.

**5.3.7** Carry out the procedure given from 5.3.1 to 5.3.6 for the dyestuff under test.

**5.3.8** Record the concentration ( $C_u$ ) of each solution of dyestuff under test from the graph prepared in 5.3.6 against their optical densities.

**5.3.9** Calculate the strength ( $S$ ) of dyestuff under test by the formula:

$$S = \frac{C_s}{C_u}$$

where

$C_s$  = concentration of solution of standard dyestuffs, and

$C_u$  = concentration of solution of dyestuff under test having same optical density as that of standard dyestuff solution.

**5.3.10** Calculate the average of all the values obtained in 5.3.9 and round off the result to the nearest whole number.

**5.3.11** If the absolute content of the standard dyestuff is known, then calculate the percentage absolute strength of dyestuff ( $S_a$ ) under test by the formula:

$$S_a = \frac{C_s \times B}{C_u}$$

where

$C_s$  = concentration of a solution of standard dyestuff,

$C_u$  = concentration of a solution of dyestuff under test having the same optical density as that of standard dyestuff solution, and

$B$  = absolute content of the standard dyestuff in percentage.

**5.3.12** For quick determination of strength of dyestuff under test with respect to that of standard dyestuff, prepare similar solution of both the dyestuffs under similar conditions and measure their optical densities as described in 5.3.4 and 5.3.5. Then calculate the strength of dyestuff under test ( $S$ ) with respect to that of the standard dyestuff by the formula:

$$S = \frac{D_1 \times Y}{D}$$

where

$D_1$  = optical density of the solution of standard dyestuff,

$D$  = optical density of the solution of the dyestuff under test, and

$Y$  = nominal strength of the standard dyestuff.

NOTE — The arithmetic mean of two parallel determinations, permissible difference between which shall not exceed 4 percent at confidence level of 95 percent shall be taken as the test result.

## 6. METHOD 2

### 6.1 Apparatus

**6.1.1 Titanium Trichloride Storage Bottle** — See Fig. 1.

**6.1.2 Reduction Flask** — See Fig. 2.

### 6.2 Reagents

**6.2.1 Hydrochloric Acid** — (a) concentrated, (b) 25 percent ( $m/v$ ).

**6.2.2 Ammonium Thiocyanate Solution** — 20 percent ( $m/v$ ).

**6.2.3 Titanium Trichloride Solution** — approximately 0.025 N, prepared as follows:

Prepare a 15 percent ( $m/v$ ) solution of titanium trichloride. Take 30 ml of solution. Filter it through a thick pad of glass wool. Add this solution to a previously boiled mixture of one litre of water and 60 ml of concentrated hydrochloric acid. Mix this solution by passing into it a current of inert gas, such as carbon dioxide or nitrogen for about 15 minutes. Store the mixture in a bottle in an atmosphere of carbon dioxide.

NOTE — Carbon dioxide may be supplied by Kipp's apparatus. The solution should be stored in a bottle painted black to protect it from light.

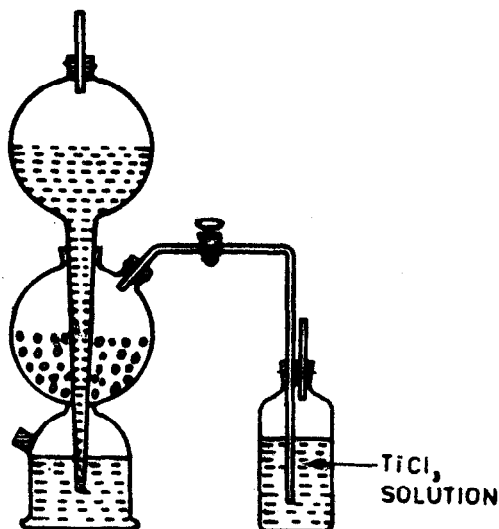


FIG. 1  $\text{TiCl}_3$  SOLUTION STORAGE BOTTLE ( PAINTED BLACK ) WITH KIPP'S APPARATUS FOR  $\text{CO}_2$  GAS

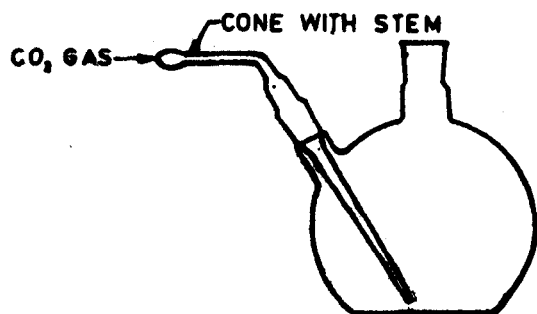


FIG. 2 500-ml REDUCTION FLASK / ( TWO NECKS, SHORT CENTRE NECK AND SIDE NECK AT AN ANGLE )

**6.2.4 Stannous Chloride Solution** — prepared by dissolving 30 g of stannous chloride in 100 ml of concentrated hydrochloric acid and diluting it to 200 ml with water. Keep the solution in air-tight bottle.

NOTE — This solution should be prepared fresh after one week.

**6.2.5 Mercuric Chloride Solution** — prepared by dissolving 5 g of mercuric chloride in 100 ml of water and filtered, if necessary.

**6.2.6 Diphenylamine Solution** — prepared by melting 1 g of diphenylamine and adding to this 100 ml of concentrated sulphuric acid. Dissolve diphenylamine by stirring.

**6.2.7 Sulphuric Acid** — 1 N.

**6.2.8 Orthophosphoric Acid** — free from iron.

**6.2.9 Potassium Dichromate Solution** — 0.1 N.

**6.2.10 Ferric Ammonium Sulphate Solution** —

Approximately 0.025 N, prepared as follows:

Dissolve 9.803 g of pure ferrous ammonium sulphate [ $\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$ ] in 100 ml of water. Add 10 ml of concentrated sulphuric acid and mix well. Dissolve 0.70 g of potassium permanganate in 100 ml of water. Add this solution to the solution of ferrous ammonium sulphate with stirring. At the end, add the permanganate solution carefully so that a ferrous salt is just oxidized. Cool the solution and dilute it to one litre. Determine the normality of the solution by the method given in Appendix A.

### 6.3 Procedure

**6.3.1** Weigh accurately 1 g of dyestuff from the test sample. Dissolve it in hot water. Pour the solution into a 500 ml graduated flask and make the volume to 500 ml. Mix well.

**6.3.2** Pipette 50 ml of the solution into a 500 ml reduction flask containing 50 ml of dilute hydrochloric acid and 50 ml of water which have been previously brought to boil. Start passing carbon dioxide into the solution and simultaneously heat the contents to boil.

**6.3.3** Pass carbon dioxide in the storage bottle containing titanium trichloride solution and immediately draw 50 ml of titanium trichloride solution by a pipette in which carbon dioxide has been passed previously. Add this solution to the dye solution (see 6.2.2). Immediately boil the contents of the flask for complete reduction (see Note 1). Cool the flask in ice-bath. Maintain the continuous flow of carbon dioxide throughout the test. Add 10 ml of 20 percent ammonium thiocyanate indicator solution. Titrate the mixture against 0.025 N ferric ammonium sulphate solution to a permanent change of colour. Note the reading of ferric ammonium sulphate solution (see Note 2).

NOTE 1 — The reduction time for various dyes ranges from 8 to 15 minutes.

NOTE 2 — The volume of ferric ammonium sulphate required for titration, should not be less than 10 ml.

**6.3.4** Carry out a blank by following the procedure given as above using 50 ml of water in place of dye solution.

**6.3.5** Calculate the titanium value, in percent, of the dye by the following formula :

$$T = \frac{(B - A) \times N \times M \times V}{E \times C \times W \times 10}$$

where

$T$  = titanium value, percent, of the dye;

$B$  = volume, in ml, of ferric ammonium sulphate solution required for blank (see 6.3.4);

$A$  = volume, in ml, of ferric ammonium sulphate solution required for the dye ( see 6.3.3 );

$N$  = normality of ferric ammonium sulphate solution ( see A-1.3 );

$M$  = molecular mass of dye;

$V$  = volume of dye solution ( see 6.3.1 );

$E$  = number of hydrogen atoms consumed per molecule of dye ( see Note );

$C$  = volume of dye solution ( see 6.3.1 ); and

$W$  = mass, in g, of the sample taken for test.

NOTE — Each azo group requires 4, nitro group 6 and hydroazo 2 atoms of hydrogen for reduction.

**6.3.6** Repeat the test prescribed in 6.3.3 twice and calculate the titanium value, in percent, of the dye.

## 7. REPORT

**7.1** Report the value obtained in 5.3.10, 5.3.11 or 5.3.12 and 6.3.5 as strength of dyestuff under test. Report also the method used, that is, Method 1 or Method 2.

## 8. SAMPLING

**8.1 Lot** — All the containers of the same dye and of the same concentration delivered to a

buyer against one despatch note shall constitute a lot.

**8.2** Unless otherwise agreed to between the buyer and the seller, the number of containers to be selected at random from a lot shall be as given below:

Lot Size	Sample Size
Up to 15	2
16 to 25	3
26 to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 and above	8

**8.3** If the dye is marketed in powder form, draw from each container as selected in 8.2 a small quantity of the dye by a suitable sampling instrument from three different parts and mix thoroughly to get a composite sample weighing about 20 g. This shall constitute the test sample.

**8.4** For dyes marketed in liquid, emulsion or paste form, shake each container as selected in 8.2 thoroughly so as to homogenize the dye dispersion and then draw the test sample as given in 8.3.

## APPENDIX A ( Clause 6.2.10 )

### DETERMINATION OF NORMALITY OF FERRIC AMMONIUM SULPHATE

#### A-1. PROCEDURE

**A-1.1** Pipette 100 ml of ferric ammonium sulphate solution ( see 6.2.10 ) into a 500 ml conical flask and add 5 ml of concentrated hydrochloric acid. Heat it to about 85°C. Add stannous chloride solution dropwise from a burette while swirling the flask over a white background until the yellow colour of the solution has nearly disappeared. Complete the reduction by adding dilute solution of stannous chloride ( see Note 1 ) dropwise with agitation after each addition, until the liquid has a faint green colour free from any tinge of yellow. Dilute the solution in the flask to thrice its volume with water. Rapidly cool the solution under the tap with stopper on and remove the excess of stannous chloride by adding 10 ml of mercuric chloride solution rapidly in one lot. A slight silky white precipitate of mercurous chloride is obtained ( see Note 2 ). Add 50 ml of 1 N sulphuric acid, 2.5 ml of orthophosphoric acid and three drops of diphenylamine solution as indicator. Titrate slowly against 0.1 N potassium dichromate solution while stirring constantly to the first permanent tinge of purple or violet blue colouration.

NOTE 1 — Dilute stannous chloride solution should be prepared by diluting stannous chloride solution ( see 6.2.4 ) with two volumes of 10 percent hydrochloric acid.

NOTE 2 — Excess of stannous chloride solution should not be added. A heavy, grey or black precipitate is the indication of too much stannous chloride solution. Discard the solution and start with a fresh solution of ferric ammonium sulphate.

**A-1.2** Carry out a blank by following the procedure prescribed in A-1.1 but by using 100 ml of water in place of 100 ml of ferric ammonium sulphate solution.

**A-1.3** Calculate the normality of ferric ammonium sulphate solution by the formula given below:

$$N = \frac{(A - B) \times N_1}{100}$$

where

$N$  = normality of ferric ammonium sulphate solution,

$A$  = volume in ml of potassium dichromate solution required to titrate the solution ( see A-1.1 ),

$B$  = volume in ml of potassium dichromate solution required to titrate the blank ( see A-1.2 ), and

$N_1$  = normality of potassium dichromate solution.



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